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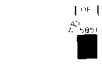

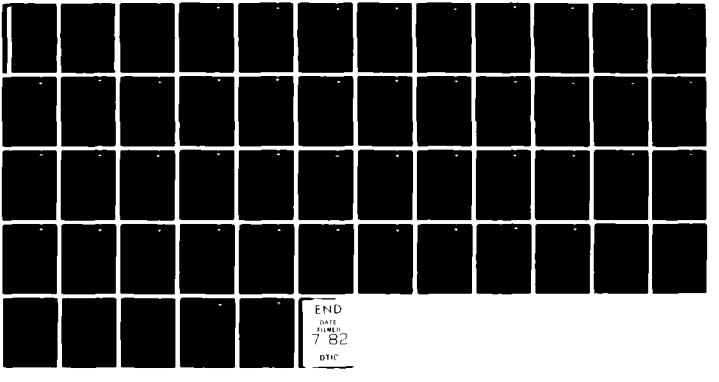

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HIGH TEMPERATURE MILLIMETER WAVE CHARACTERIZATION OF THE DIELECTRIC PROPERTIES OF ADVANCED WINDOW MATERIALS

May 1982

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FINAL REPORT

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Experimental methods have been developed to determine the dielectric properties of candidate radome materials at 35 and 94 GHz for temperatures up to 1700°C. Measurements have been carried out on fused silica, single crystal sapphire, hot-pressed silicon nitride, beryllium oxide and boron nitride as a function of temperature, composition and manufacturing procedures. Dielectric characterization data are presented in tabular and graphic forms to provide the necessary data base for evaluation of millimeter wave transmission properties of these materials in radome applications.

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1.0 ABSTRACT

Experimental methods have been developed to determine the dielectric properties of candidate radome materials at 35 and 94 GHz for temperatures up to 1700°C. Measurements have been carried out on fused silica, single crystal sapphire, hot-pressed silicon nitride, beryllium oxide, and boron nitride as a function of temperature, composition and manufacturing procedures. Dielectric characterization data are presented in tabular and graphic forms to provide the necessary data base for evaluation of millimeter wave transmission properties of these materials in radome applications.

The dielectric constants for these materials were found to be essentially independent of frequency in this region, with the loss tangents showing an increase by a factor of 2 to 4 at 94 GHz as compared to their values at 35 GHz. The observed temperature dependence of the dielectric properties indicated that significant transmission loss would occur at temperatures in excess of 1000°C for all of the ceramic materials studied.

The data obtained for hot-pressed silicon nitride strongly suggest that the observed dielectric properties are controlled by the presence and concentration of sintering aids and impurities and by microstructural properties. Potential approaches for materials improvement in terms of dielectric properties are identified for extending the useful temperature range of hot-pressed silicon nitride as millimeter wave radome materials.

2.0 SUMMARY

This report presents the results obtained from the study program, "High Temperature Millimeter Wave Characterization of the Dielectric Properties of Advanced Window Materials," AMMRC Contract No. DAAGAG-79-C-0077 covering the period 09/01/79 through 01/21/81. The long range goals of this program are to develop advanced methodology and instrumentation for determining the millimeter wave dielectric properties of candidate radome materials as a function of temperature and to determine the fundamental physical and chemical mechanisms controlling the dielectric constant and loss tangent in these materials. These candidate materials were selected to meet specific transmission as well as mechanical and thermal requirements for millimeter wave seeker window applications. The effort includes: development of methods for 35 GHz and 94 GHz measurements of dielectric properties in materials at temperatures up to 2000°C; generation of a dielectric properties data base for existing and advanced candidate materials; evaluation of these results in terms of missile sensor requirements in order to establish the design criteria and materials limitations in specific applications; and correlation of the observed dielectric properties with materials parameters. The results from the program will also facilitate new materials development by providing insight into the dependence of dielectric properties with the chemical and microstructural properties of these materials. Such understanding would allow simultaneous optimization of structural, thermal, and electromagnetic properties through control of materials parameters in order to arrive at the best overall performance for a given application.

In Phase I of the effort, covering the period of performance from 09/01/79 through 06/30/80, development of cavity spectrometer systems at 35 and 94 GHz was completed and calibration of these systems with high purity fused silica samples was successfully carried out at temperatures up to 1700°C. In addition, preliminary studies of the dielectric properties of various hot-pressed BN, Si_3N_4 and BeO were carried out at temperatures up to 1200°C, and analysis were performed to determine the required dielectric properties of the window material in terms of reflection and transmission properties for the normal incidence n-half wavelength window configurations. These results,

together with a detailed description of the experimental apparatus and methods have been reported in the interim final report, SC5235.5IRD dated February 1981.[1]

In Phase II of the effort, the following tasks have been completed:

a) Temperature studies of the dielectric properties of single crystal sapphire up to 1700°C.

b) Temperature studies of the dielectric properties of hot-pressed BN, BeO, and Si_3N_4 for various compositions and manufacturing procedures up to 1500°C at 35 GHz and 1200°C at 94 GHz.

c) Anisotropy studies of the dielectric properties of hot-pressed Si_3N_4.

d) Thermal cycling and surface contamination studies in select samples.

e) Correlation of dielectric properties of hot-pressed Si_3N_4 with composition and manufacturing process and identification of possible procedures for improvements in terms of lower absorption as well as reduced temperature dependence of its dielectric properties.

3.0 EXPERIMENTAL METHOD

The general procedures developed for measuring the dielectric properties of rod shaped samples inserted in a millimeter wave resonant cavity, as well as those for planar disk samples using a free-wave transmission and reflection method, have been described in detail in the previous report.[1] They will be summarized here for illustrative purposes.

3.1 Room Temperature Absolute Measurements

The cavity spectrometer systems are calibrated with high purity fused silica and single crystal sapphire samples by carefully studying their response (resonant frequency and quality factor Q) as a function of sample size and depth of insertion. The dielectric constants were computed from the theoretical small-sample cavity perturbation expression,

$$k = 1 + A(\Delta f/f_o)(V_o/V_s) \tag{1}$$

where k is the dielectric constant, Δf is the absolute magnitude of the resonant frequency shift from that for the empty cavity (f_o), V_o and V_s are the cavity volume and sample volume respectively, and A is a coefficient which depends on the electric and magnetic field configurations and the relative position of the sample within the cavity. The validity of the above expression is established by measuring Δf as a function of sample size. As V_s is decreased, the ratio of the observed normalized frequency shift ($\Delta f/V_s$) becomes constant, and this limiting value for k is the true dielectric constant of the material studied. The observed variations in the ratio ($\Delta f/V_s$) can be used to generate a correction curve so that the true value of k can be obtained for any given sample size over this calibration range.

In a similar manner, the dielectric loss of the sample is measured by observing the change in quality factor Q of the cavity system and using the corresponding theoretical expression,

$$\varepsilon'' = \frac{A}{2}\left(\frac{1}{Q_s} - \frac{1}{Q_o}\right)(V_o/V_s) \tag{2}$$

where Q' and Q_0 are the quality factor of the cavity with and without the sample, respectively.

This expression can be combined with Eq. (1) to give,

$$\varepsilon'' = \frac{f_o}{2(\Delta f)} \left(\frac{1}{Q'} - \frac{1}{Q_o}\right)(k - 1) \quad , \tag{3}$$

and the loss tangent can then be written as

$$\tan \delta = \frac{f_o}{2(\Delta f)} \left(\frac{1}{Q'} - \frac{1}{Q_o}\right)\left(\frac{k - 1}{k}\right) \quad . \tag{4}$$

The results obtained for the reference standards were further verified by a second independent method utilizing a plane parallel disk sample which is placed between a transmission and a receiving horn. By measuring the transmission and reflection coefficient at a given angle of incidence, k and tan δ can be calculated in a straightforward manner.

After the reference samples have been characterized, measurements are then carried out on other samples to determine the parameters $(\Delta f/V_s)$. Direct comparisons of these values with those obtained for the reference samples via the expression,

$$(k_{sample}-1)/(k_{reference}-1) = (\Delta f/V_s)_{sample}/(\Delta f/V_s)_{reference} \tag{5}$$

give the dielectric constants for the unknown samples.

Typical experimental results of the observed variation of the cavity resonant frequency in the 35 GHz system caused by the introduction of a 0.030" diameter hot-pressed Si_3N_4 sample are shown in Fig. 1. The total frequency shift Δf corresponding to the case where the sample is placed entirely through the cavity, as well as the slope in the linear region of the response curve, are directly proportional to the sample volume. The value for k obtained by comparing either of these two quantities with the corresponding values obtained for the reference samples agreed to within 0.5%, indicating excellent self-consistency for the measurement method used.

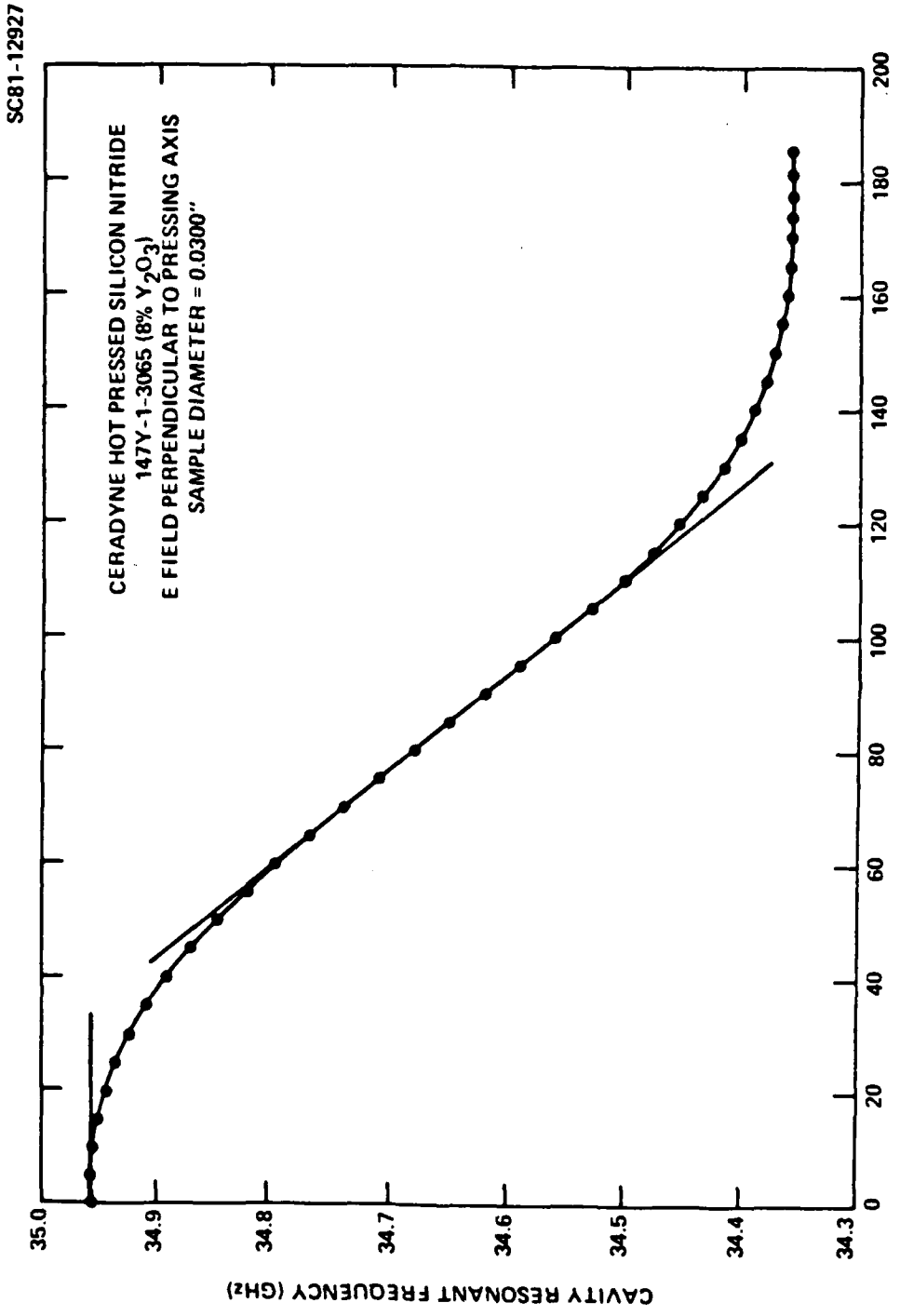

Fig. 1 Observed resonant frequency shift as a function of depth of insertion of sample into 35 GHz cavity system.

The loss tangents of the samples are determined by measuring the quantity $(1/Q' - 1/Q_o)$. These values when substituted into Eq. (4) then give the desired loss tangent for the sample.

3.2 Temperature Studies

The samples are heated in-situ, using tungsten filaments placed around the portion of the sample protruding from the cavity volume. The millimeter wave cavity systems are placed inside vacuum enclosures with viewing ports which allow observations of the samples in three directions with a visible spectra micro-optical pyrometer and an infrared pyrometer which have been calibrated for each sample material by using a known standard thermocouple and a separate heating oven. The vacuum enclosures are evacuated and filled with a reducing gas mixture (6% H_2 in N_2) to prevent oxidation of the tungsten heating elements at high temperatures. The cavity structures are maintained at near room temperature during sample heating with a circulating water system which minimizes the changes in the cavity response caused by temperature fluctuations.

The variation in the dielectric properties of the samples as a function of temperature is determined by measuring the relative changes in the cavity response from those observed at room temperature. The dielectric constants and loss tangents of the samples are calculated from the expressions,

$$(k_T-1)/(k_R-1) = (\Delta f_T/\Delta f_R) \tag{6}$$

$$\varepsilon''_T/\varepsilon''_R = [1/Q'_T - 1/Q_o]/[1/Q'_R - 1/Q_o]$$

where k_R, k_T, ε''_R, ε''_T are the values for the dielectric constant and dielectric loss at room temperature and at temperature T, and Δf_R, Δf_T, Q'_T, Q'_R, are the observed frequency shifts and the quality factors of the cavity with the sample in place at room temperature and at temperature T, and Q_o is the quality factor of the empty cavity.

A typical set of data obtained by heating an 0.030" diameter BeO sample placed in the 35 GHz cavity system is shown in Fig. 2. The observed frequency shift of the cavity resonance at room temperature for this sample is 525.1 MHz.

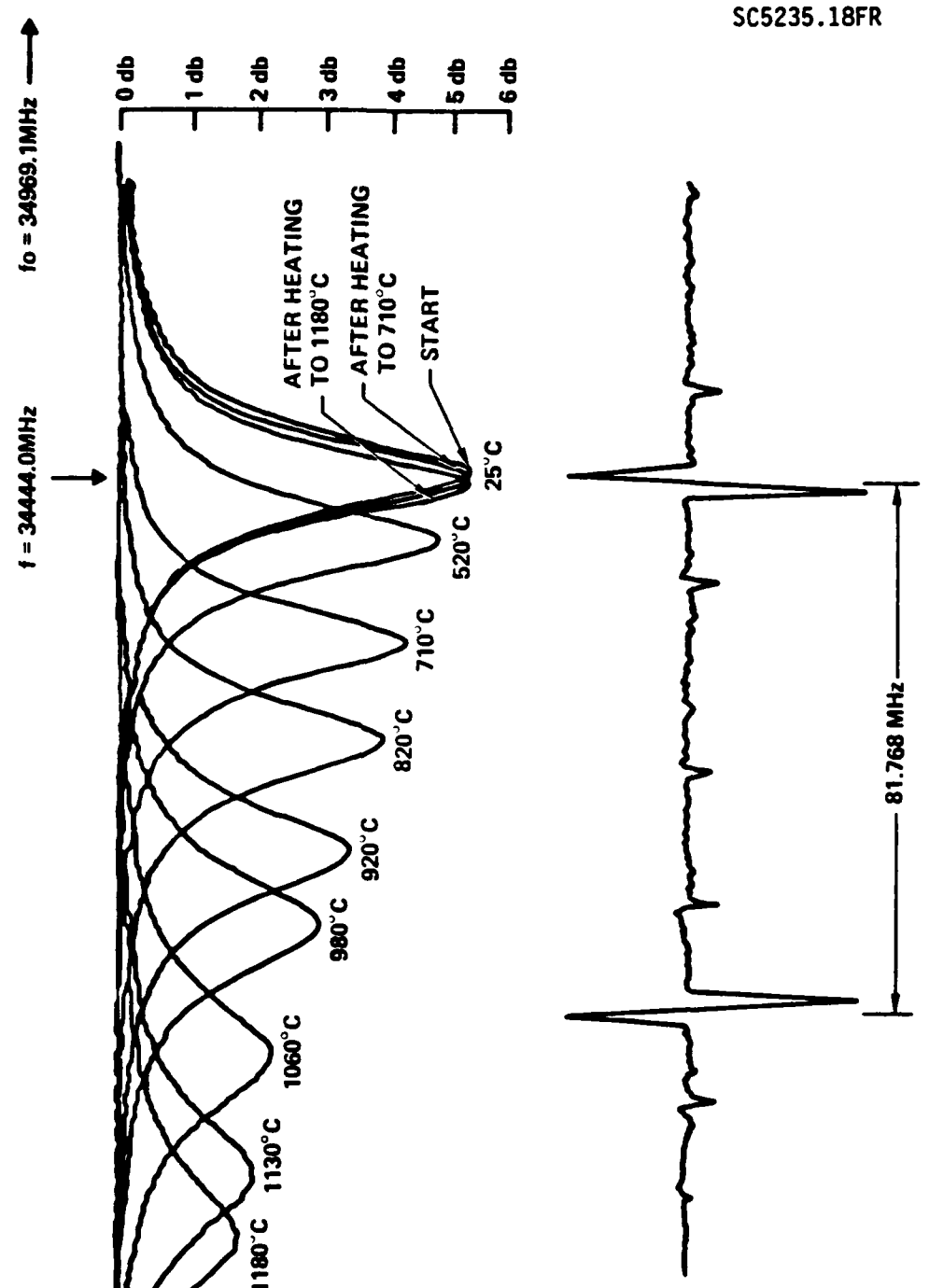

Fig. 2 Typical cavity response for 0.030" BeO sample heated to 1200°C.

Upon heating, the resonant frequency shifts to a lower value and is accompanied by a broadening of the cavity response, indicating an increase in the sample dielectric constant and loss tangent with temperature. Computation of these changes relative to the values at room temperature allow determinatin of the dielectric properties of the sample as a function of temperature.

4.0 MEASUREMENT ACCURACY AND SOURCES OF ERRORS

4.1 Measurement Accuracy

The observed reproducibility of the measurement results at 35 and 94 GHz are given in Table I. For the dielectric constant measurement, the resolution in the resonant frequency determinations are sufficiently high that the overall accuracy is primarily limited by other sources of errors, such as sample alignment and sample size. For instance, the quoted differences in accuracy for the 0.020" and 0.030" sample diameter cases are almost entirely due to the uncertainties in the sample diameter measurements.

TABLE I

SUMMARY OF MEASUREMENT ACCURACY

	35 GHz	94 GHz
Dielectric Constant Measurements		
Reproducibility on a given sample	± 0.5%	± 1%
Average over 5 samples (0.030")	± 1.4%	± 1.8%
Average over 5 samples (0.020")	± 1.8%	± 2.3%
Loss Tangent Measurements		
Typical Reproducibility	± 5%	± 10%
Average over 5 fused silica samples	± 0.0001	± 0.0003

The loss tangent measurement accuracy can generally be optimized by choosing sample sizes which give a change in the Q of the cavity by approximately a factor of 2. Under these circumstances, the reproducibility observed for a given sample is of the order of 2% at 35 GHz and 5% at 94 GHz, with an absolute detection limit of ±0.0001 at 35 GHz and ±0.0003 at 94 GHz. Typical accuracy over the entire range of observed values for the loss tangent are somewhat lower than these estimates due to the presence of systematic errors in absolute microwave power measurements, sample size, sample alignment, and sample temperature uncertainties. Consequently, the overall accuracy is conservatively taken to be ±5% at 35 GHz and ±10% at 94 GHz, respectively.

4.2 Systematic Errors Caused by Sample Heating

The three primary systematic sources of errors in the measurements cauesd by sample heating are:

a. Change in cavity characteristics caused by the residual increase in cavity wall temperature as a consequence of sample heating.

b. Temperature gradient in sample.

c. Thermal expansion of the sample during the heating cycle.

Since the temperature of the cavity structure cannot be held rigorously constant during a sample heating cycle, the resonant frequency as well as the quality factor of the empty cavity will change as a function of sample temperature. Consequently, these changes need to be taken into account when the measured dielectric properties are computed via Eq. (6). The observed cavity temperature change as a function of approximate sample temperature is shown in Fig. 3. The increase in cavity temperature varies approximately as the square of the sample temperature and is of the order of +4°C and +16°C at sample temperatures of 800°C and 1600°C respectively. The observed cavity resonant frequency change as a function of cavity temperature is also shown in the figure; these are approximately -2 MHz and -8 MHz at sample temperatures of 800°C and 1600°C. Since the typical measured values of Δf in Eq. (6) are of the order of several hundred megacycles, these corrections are at most a few percent and can be accounted for to within 0.1 to 0.2 percent by monitoring the cavity wall temperature during each temperature run. A similar study of the change in quality factor of the empty cavity as a function of cavity temperature was also performed and showed that corrections with accuracy of the order of a few tenths of percent can be made over the entire temperature range. Consequently, the errors introduced in the dielectric constant and loss tangent measurements by this effect are essentially negligible when compared to the experimental accuracies stated in Table I.

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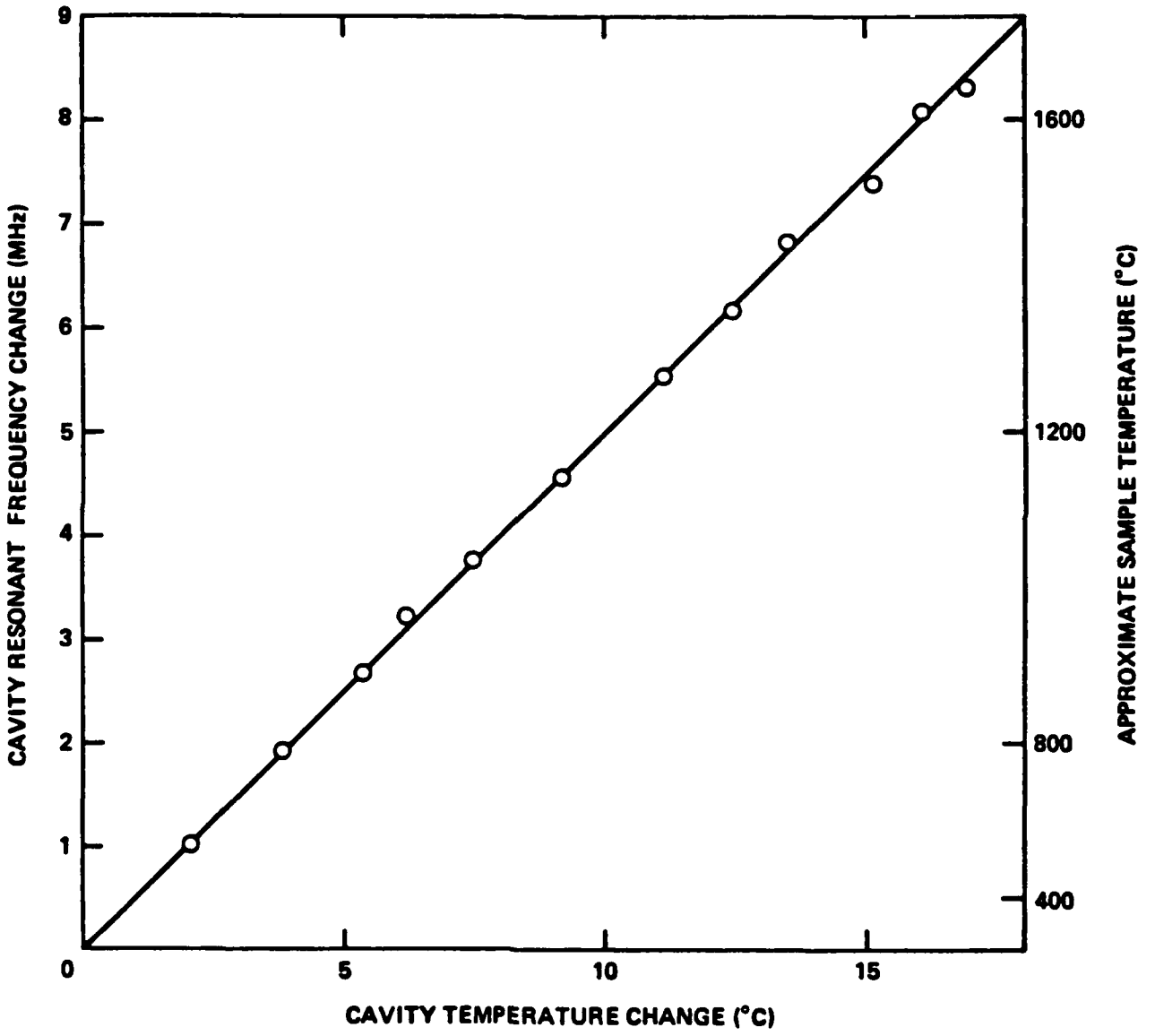

Fig. 3 Observed cavity resonant frequency change as a function of cavity temperature and approximate sample temperature.

Since the samples are heated only at the ends rather than uniformally throughout, substantial temperature gradients can occur, particularly at high temperatures. The measured temperature gradient as a function of the observed temperature at the center of the sample is shown in Fig. 4. At high sample temperatures, the observed gradients are approximately linear with the temperature measured at the center of the sample and have magnitudes on the order of ±70°C and ±150°C at 1000°C and 1600°C, respectively. Although these gradients appear to be quite large in terms of absolute values, they are not significant in terms of their influence on the measurement accuracy of the dielectric constant. This is due to the fact that the temperature dependence of the dielectric constants for all of the samples studied in this program are rather small. With the exception of single crystal sapphire and hot pressed BeO, which showed an increase in k by approximately 20% from 25°C to 1600°C, the variations observed are typically less than 10% over this temperature range. In addition, these changes are fairly gradual so that over any given temperature interval of the order of 200°C, the variation of k with temperature is essentially linear. Consequently, the measured k as deduced via Eq. (6) is properly weighted and corresponds to the correct value for the average temperature of the samples. Therefore, the uncertainty introduced by these gradients is at most of the order of a few tenths of a percent.

Although the measured loss tangents are strong functions of temperature for the hot-pressed ceramic samples, their behavior showed two fairly linear response regions with a sharply varying transition region occurring in the temperature interval from 700°C to 1100°C. Over the linear portions of these curves, Eq. (6) gives the proper average values so that the effects caused by the temperature gradients in the samples are automatically accounted for. In the transition region, the non-linearity of the loss tangent with temperature could cause significant errors in the measurements. Depending on how fast the slopes are changing, errors upward of several percent could result; this is the primary reason for quoting an overall error in Table I which is twice that for the observed reproducibility in the microwave measurement of the loss tangent measurements.

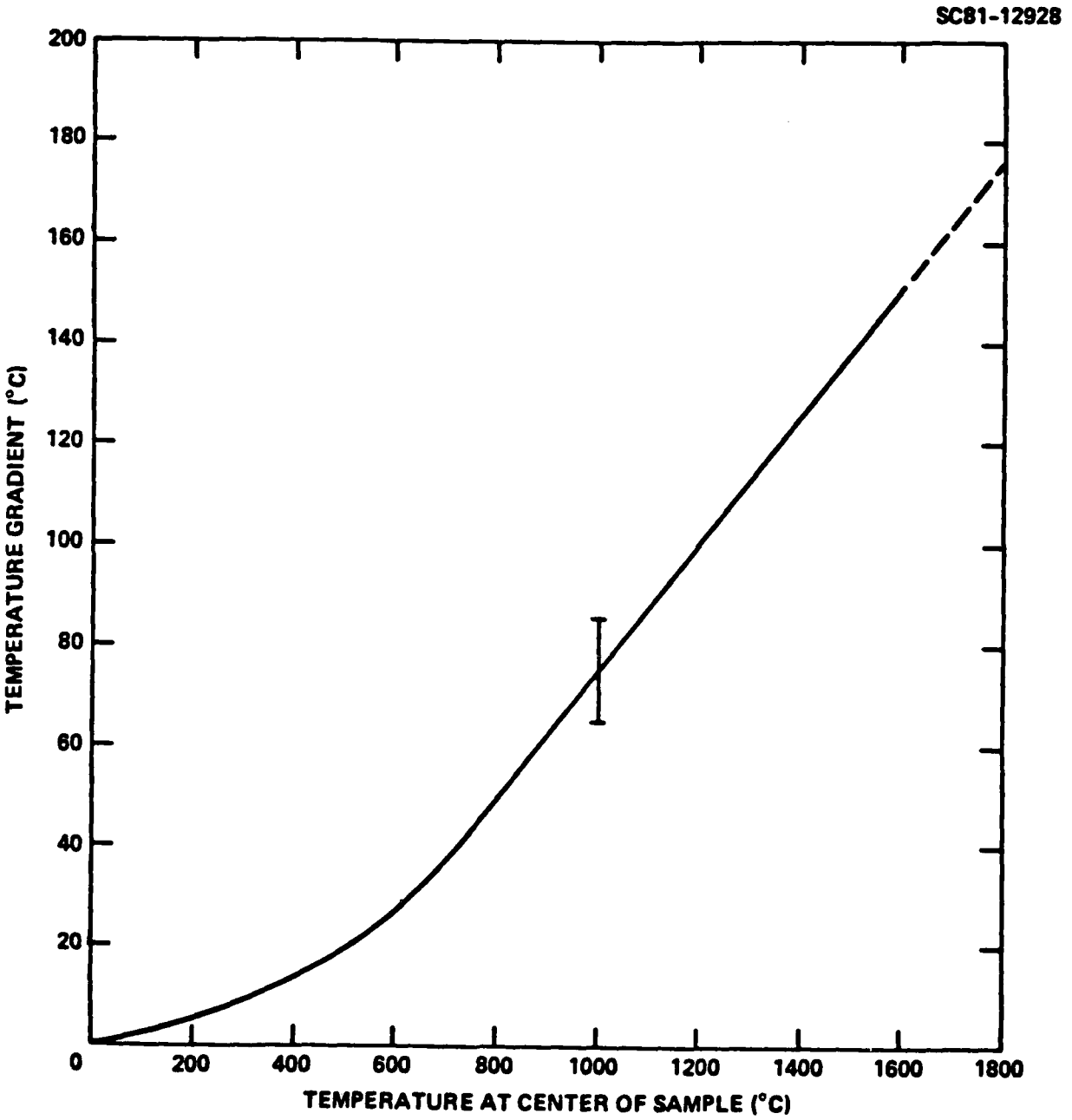

Fig. 4 Measured temperature gradient in samples as a function of average sample temperature.

The third source of measurement error caused by sample heating is that associated with thermal expansion of the sample during the heating cycle. For the 35 GHz measurements, where the sample is placed entirely through the cavity, this effect is expected to be negligible since the total mass of the sample contained within the cavity volume is essentially constant as a function of temperature. Consequently, corrections need not be applied to deduce the true value of k or loss tangent at a given temperature. For the 94 GHz measurements, where the geometry of the system restricts the placement of the sample to only partially contained within the cavity volume, thermal expansion in the linear dimension of the rod shaped samples causes additional mass to be introduced into the cavity. Consequently, the sample volume term (V_s) in Eq. (5) is no longer constant as a function of temperature, and the measurements of Δf and Q' need to be corrected for sample size changes at each temperature of observation. For those cases where the thermal expansion coefficients of the samples are less than $10^{-6}/°C$ (fused silica, single crystal sapphire), the change in the length of sample contained within the cavity volume is only of the order of 0.2% or less at temperatures up to 2000°C. Since the observables used to deduce the dielectric properties (Δf and Q) are linear functions of the sample length, this value corresponds to an error in the measured changes in dielectric properties ($\Delta\varepsilon'/\Delta T$, $\Delta\varepsilon''/\Delta T$) as a function of temperature of 0.2%. Consequently, the errors introduced for these low expansion coefficient samples are negligible in the determination of both k and the loss tangent of the samples at high temperatures. For samples with thermal expansion coefficients of the order of $10^{-5}/°C$ or larger, these errors exceed several percent, and therefore need to be corrected for. Since the thermal expansion coefficients for the ceramic samples are generally not accurately known over the wide temperature range of this study, experiments were carried out to generate the necessary coefficients for each sample type to correct the observed data at 94 GHz. This is accomplished by inserting the samples partially into the 35 GHz cavity and observing the resulting change in the cavity resonant frequency shift as a function of sample temperature. The ratio of the frequency shift at temperature a, to that observed at temperature b, can then be expressed as,

$$(\Delta f)_a/(\Delta f)_b = (k_a-1)/(k_b-1) + (\Delta \ell/\ell) \qquad (7)$$

where k_a and k_b are the dielectric constants at temperatures a and b previously determined with the sample placed entirely through the cavity, and $\Delta \ell/\ell$ is the fractional change in sample length caused by thermal expansion.

The values for $(\Delta \ell/\ell)$ determined by this method is then subtracted from the frequency ratio $(\Delta f)_a/(\Delta f)_b$ measured at 94 GHz in order to compute the dielectric constant ratio $(k_a-1)/(k_b-1)$ at the two temperatures a and b.

5.0 ROOM TEMPERATURE RESULTS

5.1 Reference Standards

The results obtained at room temperature (22°C) for the dielectric properties of high purity fused silica (Dynasil 4000) and single crystal sapphire (Tyco Sapphicon) at two different crystal orientations with respect to the electric field are shown in Table II. The dielectric properties were measured by both the cavity perturbation and by the free-space transmission and reflection methods. For the cavity perturbation method, samples varying in diameter from 0.010" to 0.060" with three to five samples at each diameter were used. For the free space method, planar circular disk samples 3" in diameter with thicknesses ranging from 1/8" to 1/2" were used. The agreements in the measured results were found to be consistent with the experimental errors given in Table I. The dielectric constants of the samples are essentialy independent of frequency to within the experimental error between 35 GHz and 94 GHz, while the loss tangents increased by approximately a factor to 2.

The measured dielectric properties for Dynasil 4000 at 35 GHz are in excellent agreement with the values reported by Westphal[2] over the same frequency region. The dielectric constants obtained for single crystal sapphire are in good agreement with the values reported by Westphal and Sils[3] at 8.5 GHz, while the loss tangents are substantially higher than the reported value for high-purity (Union Carbide) samples. They are, however, in general agreement with the values measured by these same authors for Linde Air Product single crystal sapphire samples.

5.2 Hot-Pressed Boron Nitride and Beryllium Oxide

Boron nitride samples were obtained from Carborundum Co. (Grade HP) and Union Carbide (Grade HBC) and machined into cylindrical rods of approximately 1" in length and 0.030" to 0.040" in diameter. For both cases, the axes of the rod-shaped samples were along the direction of the pressing axis so that when placed inside the cavity resonators, the electric fields were oriented parallel to the pressing axis. The density of the Carborundum HP samples were found to

TABLE II

MEASURED ROOM TEMPERATURE DIELECTRIC PROPERTIES OF REFERENCE STANDARDS

Sample	Density	35 GHz K	35 GHz Tan δ	94 GHz K	94 GHz Tan δ
Dynasil 4000	2.195	3.82	0.0004	3.78	0.0006
Sapphire, Optical Axis at 0° to E field	3.97	11.72	0.0043	11.63	0.0088
Sapphire, Optical Axis at 60° to E field	3.94	9.81	0.0066	9.67	0.012

be slightly less than that for the Union Carbide HBC samples, and a corresponding difference of approximately 5% in the dielectric constants was observed. The loss tangents, however, differed by a factor of 4, with the Union Carbide sample showing a lower value. As in the previous case for the reference standards, the dielectric constants were essentially independent of frequency between 35 GHz and 94 GHz. However, the loss tangents showed an increase by a factor of 2 to 3.

The beryllium oxide samples used in this study were obtained from the National Beryllia Company (K150) and from Ceradyne Corp. (418S). The samples used were 0.030" in diameter. The measured dielectric properties were almost identical at the two frequencies of observation, with the loss tangents showing an increase of a factor of 3 between 35 GHz and 94 GHz.

The data obtained for hot-pressed boron nitride and beryllium oxide at room temperature are shown in Table III.

5.3 Hot-Pressed Silicon Nitride

The room temperature dielectric properties of hot-pressed silicon nitride were studied as a function of sample composition, different fabrication procedures, and orientation of sample pressing axis with respect to the incident electric field. The samples were obtained from the Ceradyne Corporation in the

TABLE III

MEASURED ROOM TEMPERATURE DIELECTRIC PROPERTIES OF HOT PRESSED BN AND BeO

Sample	Density	35 GHz K	35 GHz Tan δ	94 GHz K	94 GHz Tan δ
BN, Carborundum HP (E field II to pressing axis)	1.98	4.23	0.0023	4.20	0.0052
BN, Union Carbide HBC (E field II to pressing axis)	2.01	4.45	0.0006	4.48	0.0015
BeO, National Beryllia K150	2.85	6.80	0.0010	6.75	0.0033
BeO, Ceradyne 418S	2.86	6.61	0.0012	6.70	0.0038

forms of precision centerless-ground 1" cylindrical rods ranging in diameter from 0.020" to 0.060". An average of 3 to 5 samples at each diameter were used to study the homogeneity in dielectric properties within each sample type. The maximum variations observed for the dielectric constants and loss tangents were respectively, ±2% and ±3% in k at 35 GHz and 94 GHz and ±10% and ±15% for the loss tangent at 35 GHz and 94 GHz. The average values obtained for the various samples at 35GHz are shown in Table IV.

As can be seen, there are considerable differences in the observed dielectric properties for samples with different composition. The dielectric constant ranged from a low value of 8.12 for a 5% MgO sample to a high value of 9.66 for the 15% Y_2O_3 sample. The loss tangents showed differences of over an order of magnitude between a 8% Y_2O_3 sample and the 15% Y_2O_3 sample. As expected, there is a correlation between sample density and dielectric constant, while the loss tangents are generally smaller for samples having lower concentrations of sintering aids. In addition, both the dielectric constants and the loss tangents are larger for a given sample with the electric field oriented parallel to the pressing axis as compared with their values for the electric field aligned perpendicular to the pressing axis.

TABLE IV

MEASURED ROOM TEMPERATURE DIELECTRIC PROPERTIES OF HOT PRESSED SILICON NITRIDE AT 35 GHz

Sample Composition	Designation	Density	K	Tan δ	Remarks
1. 1% MgO	147A(3553-1)	3.18	8.25	0.0036	Tungsten carbide ball mill.
2. 1% MgO	147A(5354-1)	3.14	8.62	0.011	Tungsten carbide ball mill.
3. 5% MgO	147L(5456-2)	3.21	8.94	0.0078	β-Si_3N_4 used as starting mat.
4. 5% MgO	147L(5456-1)	3.19	9.12	0.0147	β-Si_3N_4 used with different processing.
5. 5% MgO	147E(5481-1)	3.13	8.12	0.0083	α-Si_3N_4 from different supplier.
6. 8% Y_2O_3	147Y-1(3065)	3.21	8.59	0.0033	E field parallel to pressing axis field.
7. 8% Y_2O_3	147Y-1(3065)	3.23	8.29	0.0015	E field perpendicular to pressing axis
8. 8% Y_2O_3	147Y-1(5156)	3.34	9.44	0.0107	
9. 8% Y_2O_3	147Y-1(3964-2)	3.27	8.60	0.0057	
10. 15% Y_2O_3	147Y(1846)	3.29	9.66	0.0195	

All samples except (6) are with E field perpendicular to pressing axis.

The data obtained at 94 GHz for select samples are shown in Table V. As in the previous cases for other materials, the primary difference between the dielectric properties at 35 and 94 GHz is an increase in the loss tangent by a factor of 2 to 3.

TABLE V

MEASURED ROOM TEMPERATURE DIELECTRIC PROPERTIES OF HOT-PRESSED SILICON NITRIDE AT 94 GHz

Sample Composition	Designation	Density	K	tan δ
1. 1% MgO	147A(3553-1)	3.18	8.28	0.012
2. 5% MgO	147L(5456-2)	3.21	9.01	0.024
3. 8% Y_2O_3	147Y-1(3065)	3.23	8.22	0.006
4. 15% Y_2O_3	147Y(1846)	3.29	9.70	0.064

All samples are with E field perpendicular to pressing axis.

6.0 THERMAL CYCLING & MOISTURE TEST RESULTS

Two types of thermal cycling tests were carried out to determine whether any non-reversible changes in the dielectric properties caused by exposure of the samples to high temperature environments are present. In the first case, the dielectric properties of the samples were measured at room temperature. The samples were then removed from the measurement apparatus and placed in an oven and heated to about 1000°C for periods up to several hours. The samples were then cooled to room temperature and their dielectric properties remeasured and compared to the values initially obtained. In the second case, the samples were mounted in the measurement apparatus and maintained under a reducing atmosphere while the sample temperature was cycled by rapidly changing the current flowing through the sample heating coil. An illustration of this latter case is shown in Fig. 2 for a 0.030" beryllium oxide sample where the cavity responses at room temperature are shown after the sample has been heated to 710°C and 1180°C. The changes in the dielectric properties of the sample were then determined by comparing these cavity responses with the responses obtained initially before heating.

For the reference standards, i.e, high purity fused silica and single crystal sapphire, the results obtained indicated that there were no measurable changes in their dielectric properties due to thermal cycling at temperatures up to the capability limits for the two methods used (1300°C in air and ~ 1600°C in the reducing atmosphere).

As indicated by Fig. 2, an initial thermal cycling for beryllium oxide at 1180°C caused an increase in the dielectric constant by approximately 1 to 2%, while the dielectric loss remained essentially constant. Subsequent sample heating to 1180°C and cooling produced no further additional changes. In view of the relatively small magnitude of this change, it is difficult to determine its exact dependence on final sample temperature, but the result obtained for heating to 710°C indicated that the effect increases with temperature.

Tests were carried out with both methods on hot-pressed silicon nitride samples. Samples exposed to air and heated to 1300°C in an oven for periods up to one hour produced negligible changes in both the dielectric constant and the loss tangent. On the other hand, rapid heating to 1400°C and cooling to room temperature in a reducing atmosphere caused a decrease in the observed dielectric constant by abut 1 to 2%, while the loss tangent increased by approximately 5%.

It is not possible at present to identify the mechanisms causing these effects, but their magnitudes are of the same order as the observed variations in room temperature dielectric properties for different samples of the same material and composition, and are much smaller than the observed temperature dependence of the dielectric properties. Consequently, they are not large enough to be of significance in the determination of transmission properties for these materials as a function of temperature.

7.0 TEMPERATURE STUDIES

7.1 Reference Standards

The dielectric properties of Dynasil 4000 fused silica as a function of temperature at 35 and 94 GHz are given in Tables VI and VII. The results obtained at 35 GHz are in excellent agreement with the data previously reported by Westphal.[2] The dielectric constant increases slowly with temperature while the loss tangent is essentially independent of temperature to within the quoted experimental accuracy over the temperature range studied. Very similar behaviors are observed at 94 GHz, with the exception of the loss tangents being approximately a factor of 2 larger at the higher frequency.

The dielectric properties of single crystal sapphire (Tyco Sapphicon) as a function of temperature at 35 GHz and 94 GHz are shown in Figs. 5, 6, 7, and 8. Both the dielectric constant and the loss tangent are strongly dependent on temperature at the frequencies of observation. At 35 GHz, the increase in dielectric constant at 1600°C is approximately 20% while the corresponding increase in the loss tangent is approximately a factor of 3. Variations of the same order of magnitudes are observed at 94 GHz.

7.2 Hot-Pressed Boron Nitride and Beryllium Oxide

The results obtained for the dielectric properties of hot-pressed boron nitride samples as a function of temperature at 35 GHz are shown in Figs. 9 and 10. Similar results for beryllium oxide samples are shown in Figs. 11 and 12. The dielectric constants for boron nitride exhibit a relatively slow increase in their values with increasing temperature. By contrast, an increase of approximately 30% is observed for the beryllium oxide samples from room temperature to 1400°C. For both types of samples, there is a rapid increase in their loss tangents at temperatures in excess of 1000°C.

Similar behavior in the dielectric properties for these samples is observed at 94 GHz. These results are given in Figs. 13 and 14.

TABLE VI

SUMMARY OF DIELECTRIC DATA FOR DYNASIL 4000 TAKEN AT 35 GHz

Current Result			Westphal Data (35.1 - 37.3 GHz)		
Temperature	K	Loss Tangent × 10^4	Temperature	K	Loss Tangent × 10^4
25	3.82 (0.02)	4.0 (1)	23	3.82 (0.015)	3.5 (1)
100 (5)	3.82	3.5			
200 (5)	3.84	3.0	195	3.84	3.0
300 (5)	3.85	3.0			
400 (10)	3.87	3.0	403	3.86	3.0
600 (15)	3.88	3.0	610	3.87	2.0
800 (20)	3.90	3.0	807	3.89	2.0
1000 (40)	3.93	3.5	1003	3.92	3.0
1200 (50)	3.96	3.5	1205	3.95	3.0
1400 (50)	3.98	6.0	1408	4.01	4.0
1625 (60)	4.16	12.0	1650	4.14	10.0

Free Wave Results

25	3.83 (0.002)	Less than 7.0

TABLE VII

SUMMARY OF DIELECTRIC DATA FOR DYNASIL 4000 TAKEN AT 94 GHz

Cavity Method			Free Wave Method	
Temperature	K	Loss Tangent	K	Loss Tangent
25	3.78 (0.04)	0.0006 (2)	3.81 (0.04)	Less than 0.001
100 (5)	3.78	0.0006		
200 (5)	3.80	0.0005		
300 (10)	3.82	0.0005		
400 (10)	3.83	0.0005		
600 (15)	3.86	0.0005		
800 (25)	3.89	0.0007		
1000 (50)	3.91	0.0008		
1200 (80)	3.94	0.0009		

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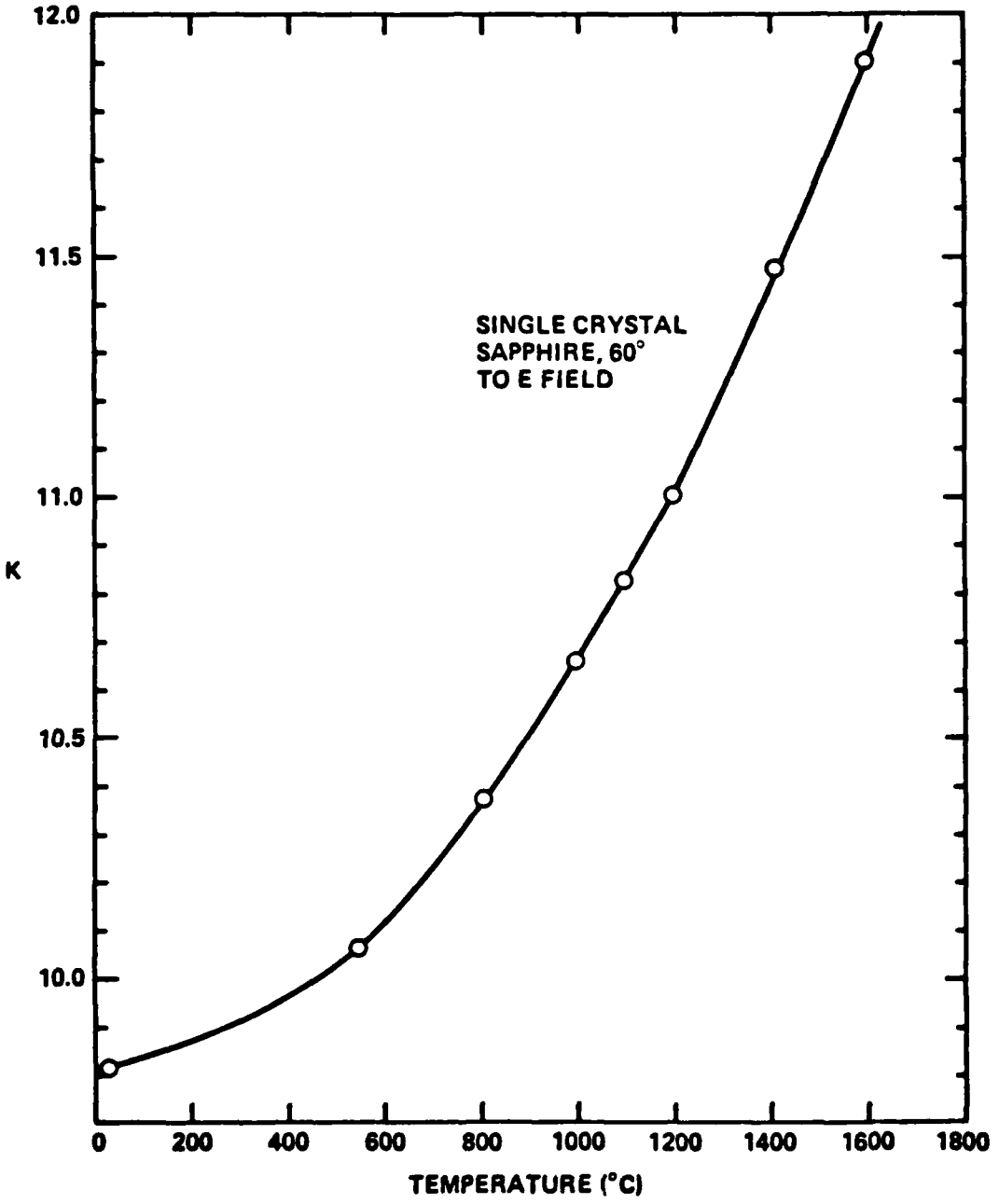

Fig. 5 Measured dielectric constant of single crystal sapphire at 35 GHz (crystal axis 60° to E field).

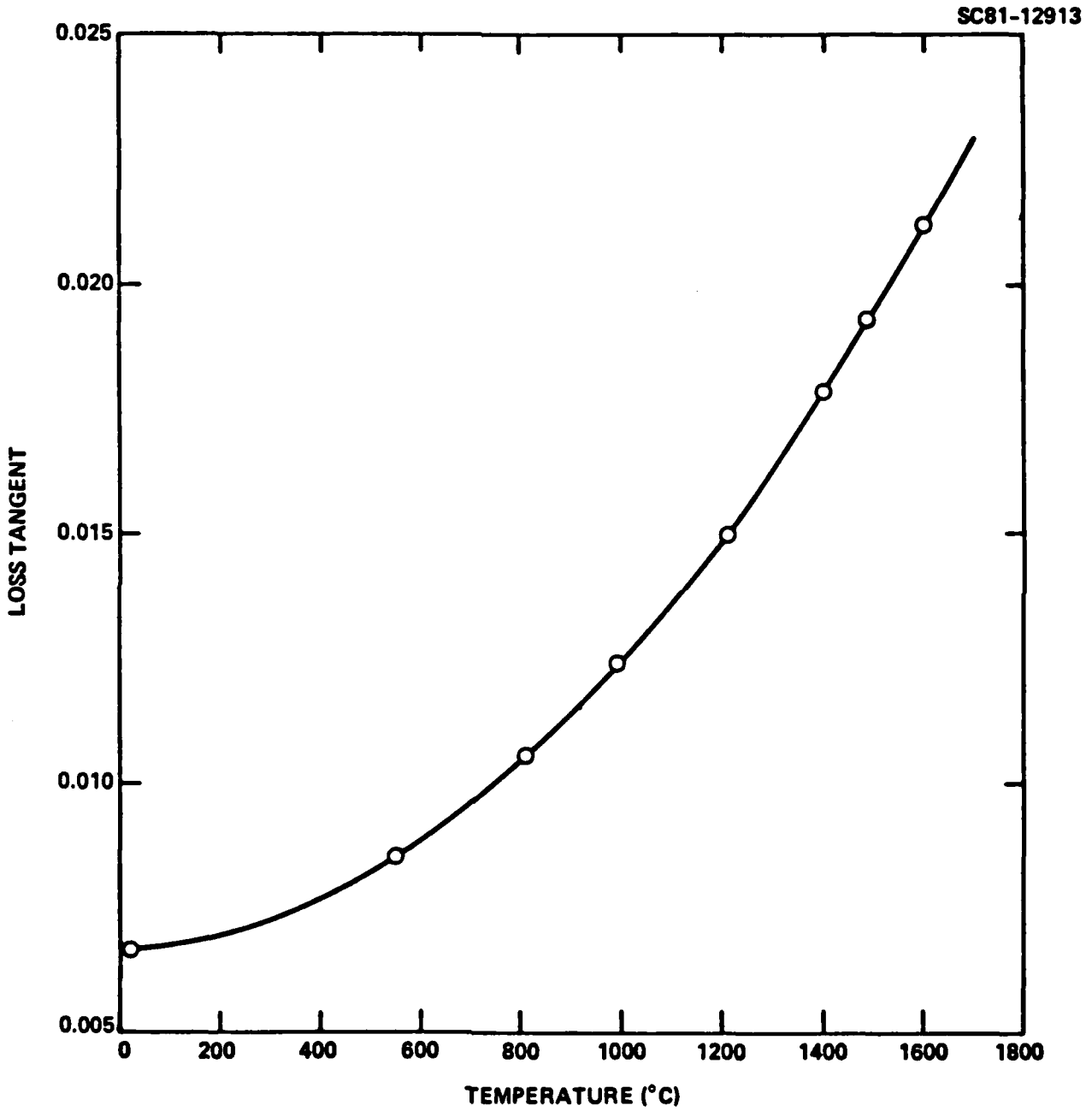

Fig. 6 Measured loss tangent of single crystal sapphire at 35 GHz.

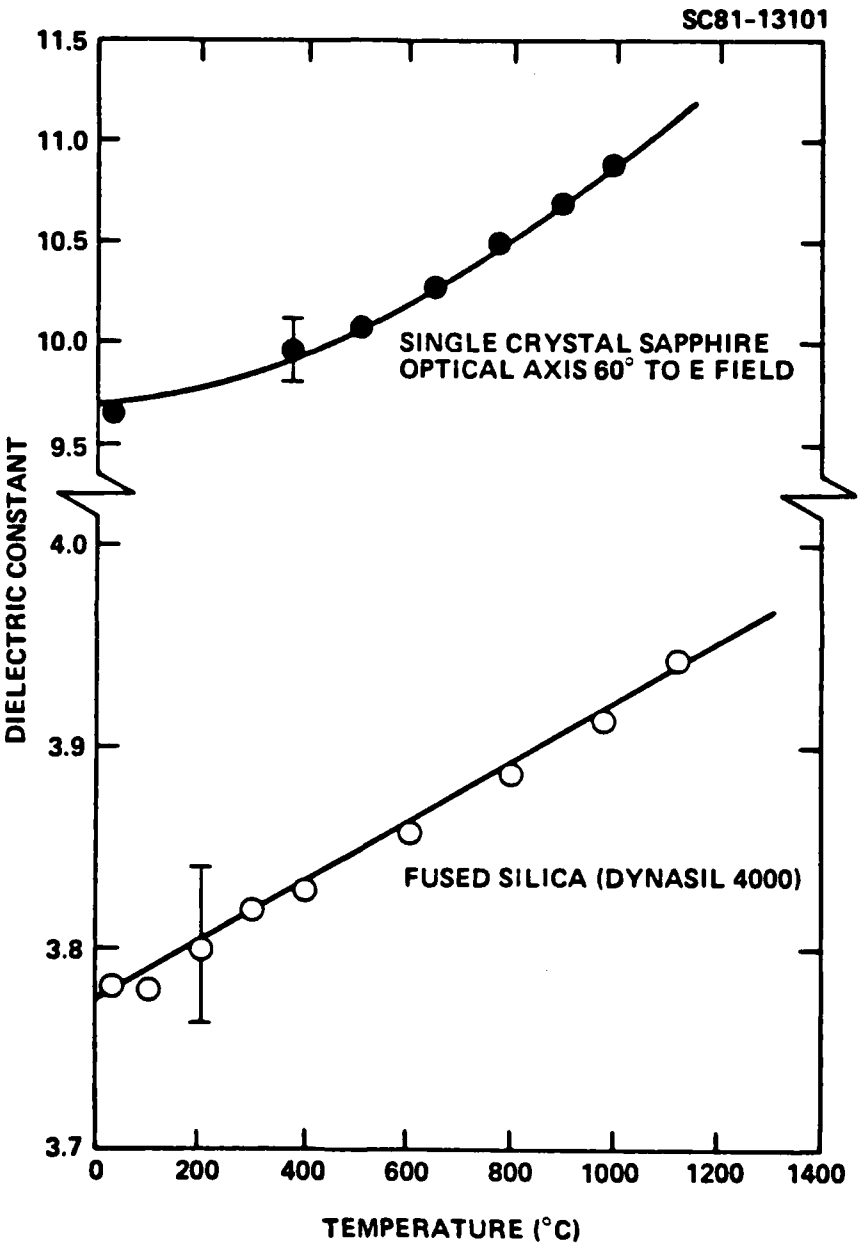

Fig. 7 Measured dielectric constants of reference standards at 94 GHz.

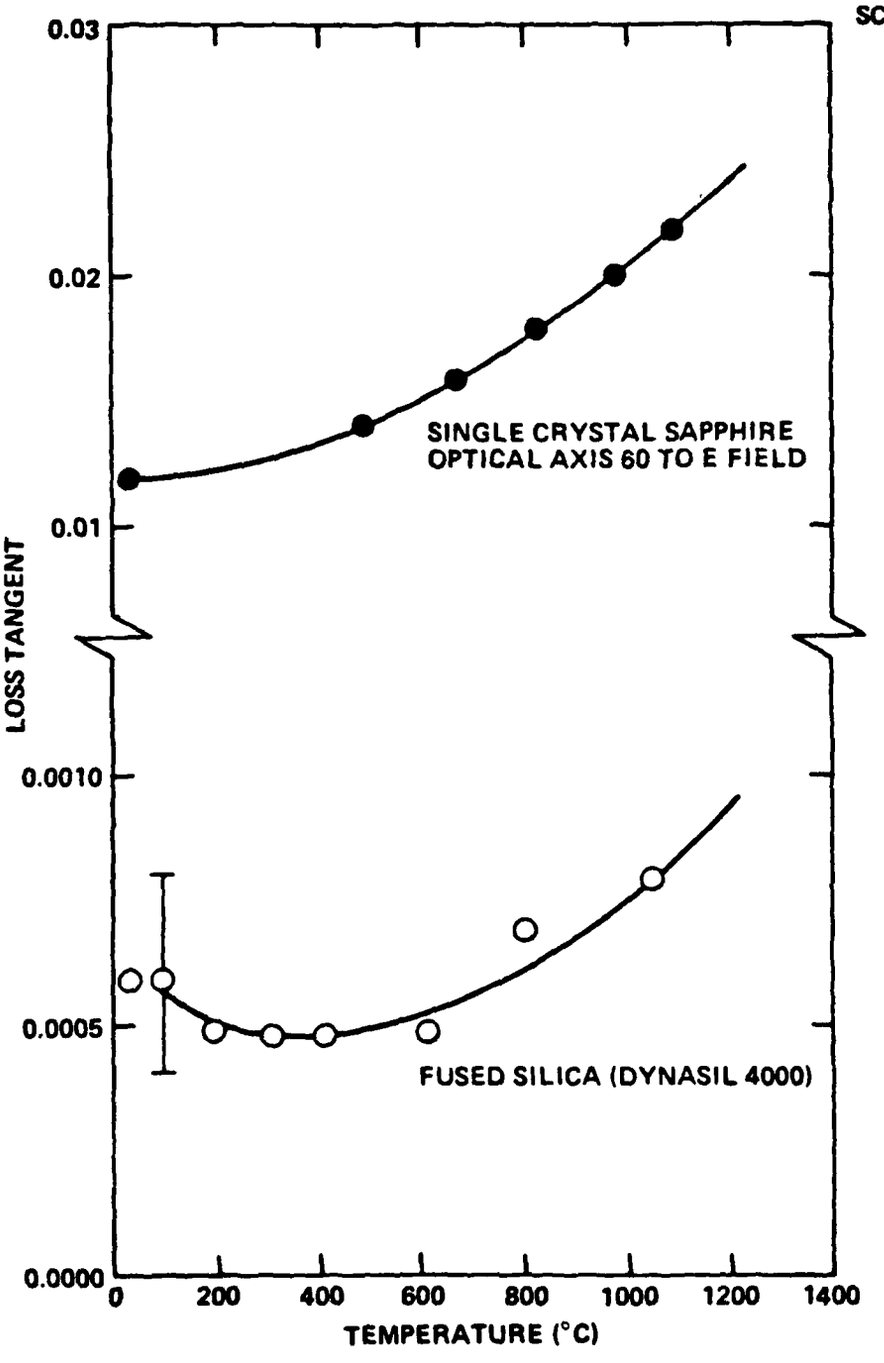

Fig. 8 Measured loss tangents of reference standards at 94 GHz.

SC81-12918

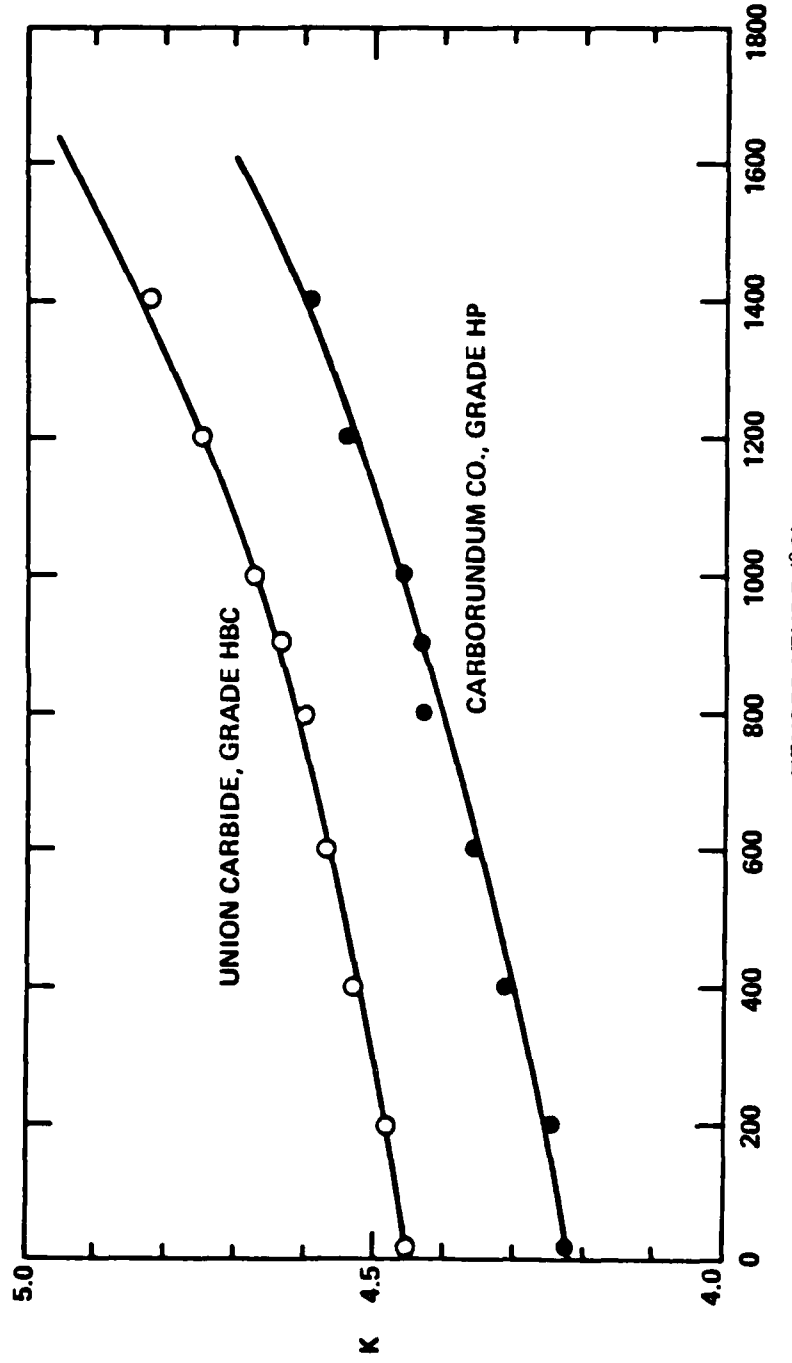

Fig. 9 Measured dielectric constant of hot-pressed boron nitride at 35 GHz.

SC81-12917

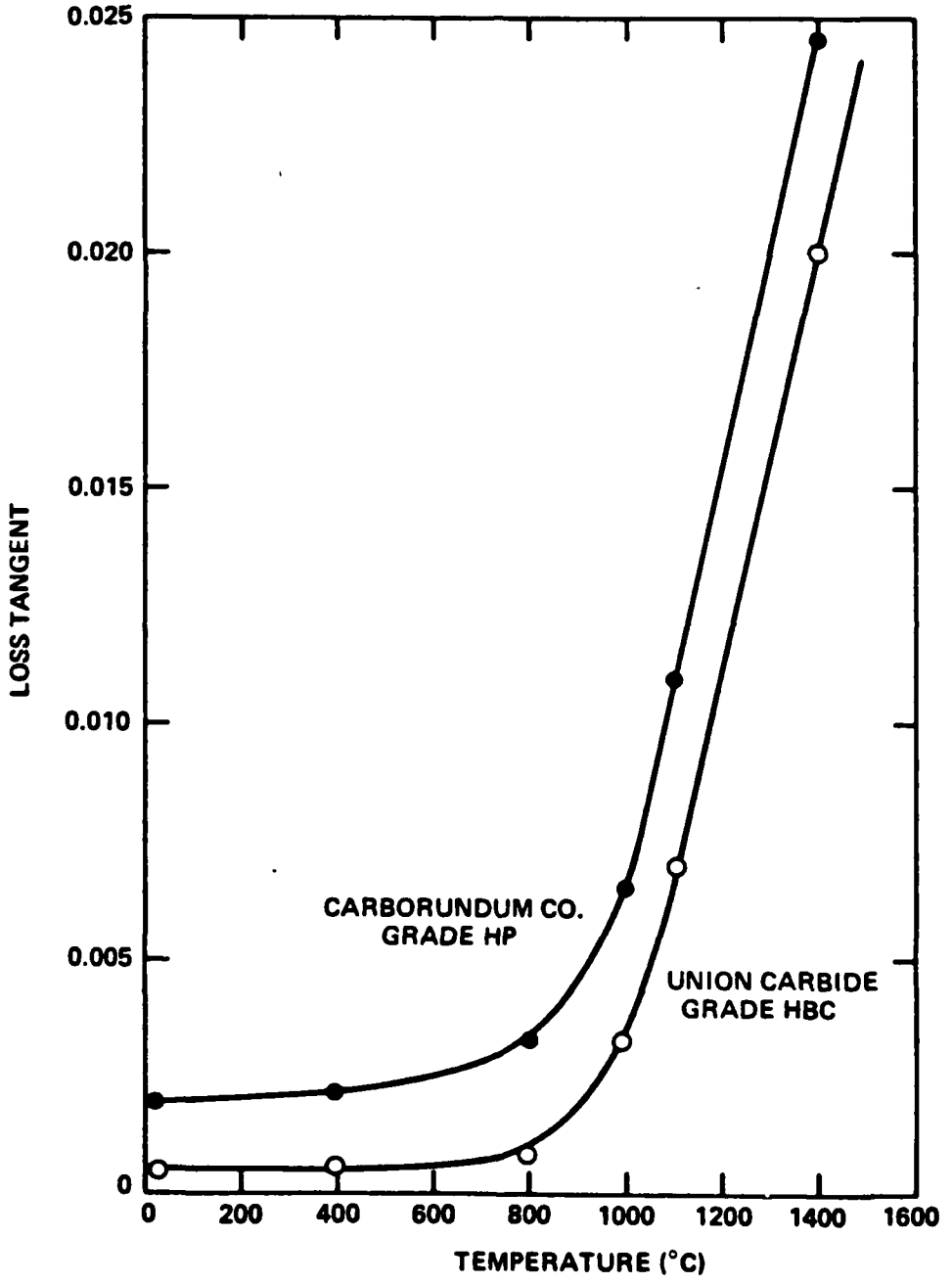

Fig. 10 Measured loss tangent of hot-pressed boron nitride at 35 GHz.

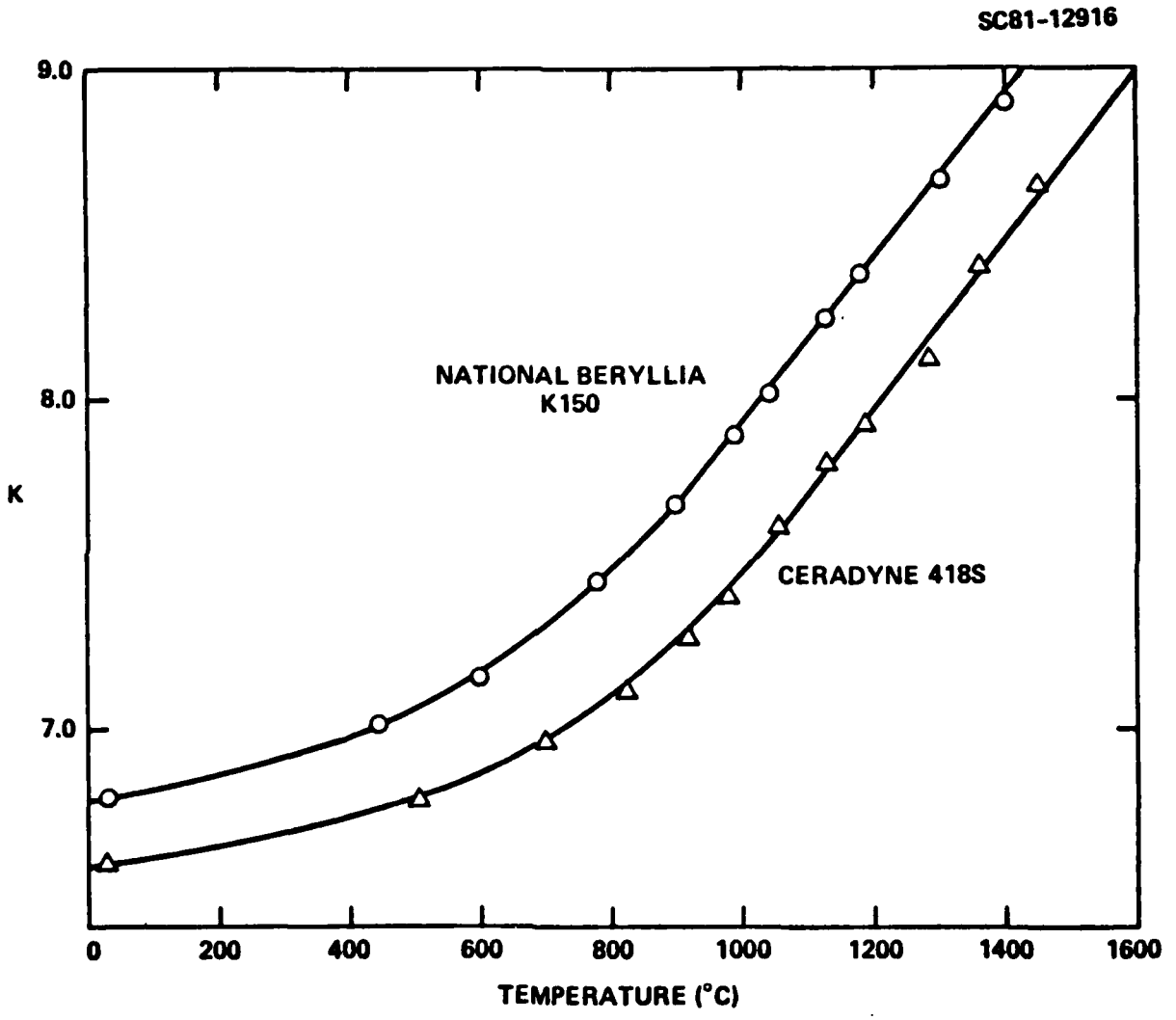

Fig. 11 Measured dielectric constant of beryllium oxide at 35 GHz.

SC81-12914

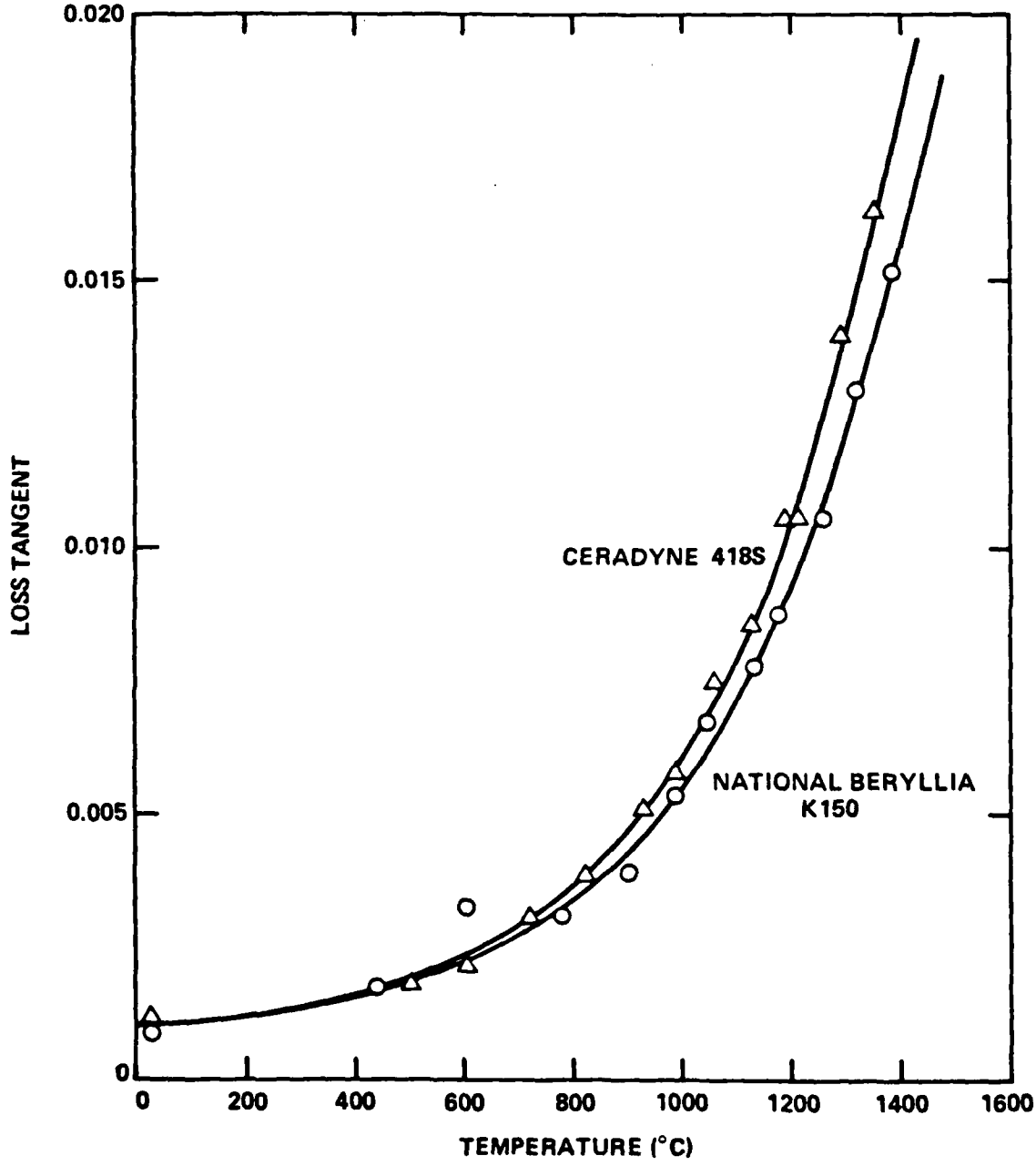

Fig. 12 Measured loss tangent of beryllium oxide at 35 GHz.

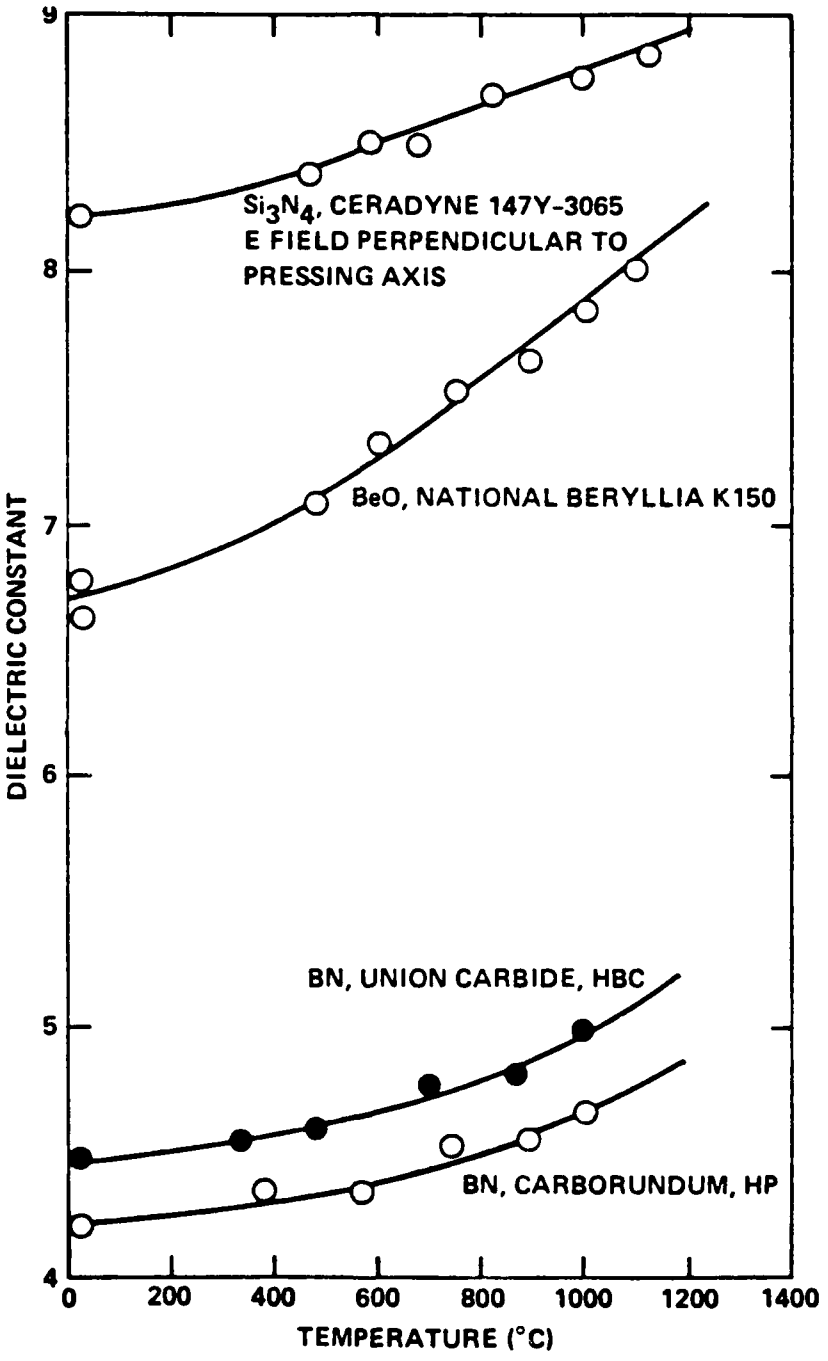

Fig. 13 Measured dielectric constants of samples at 94 GHz.

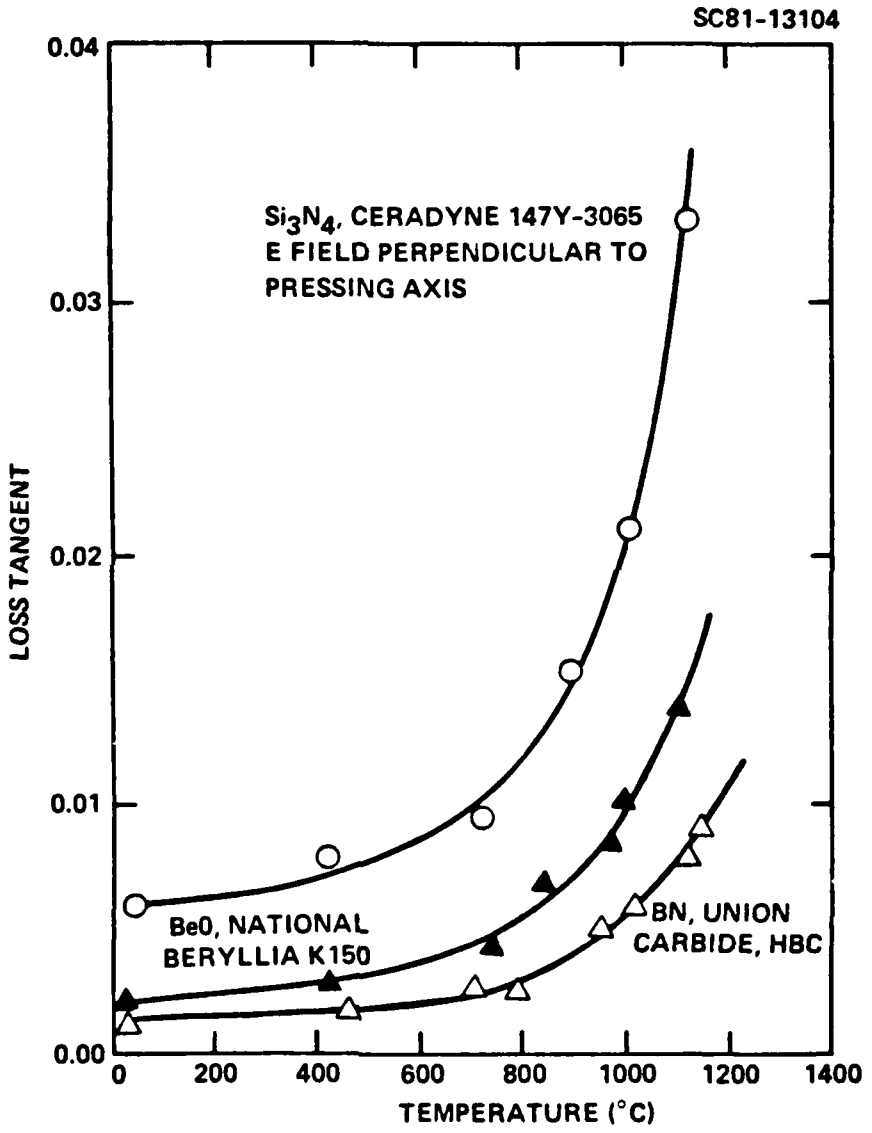

Fig. 14 Loss tangents of samples at 94 GHz.

7.3 Hot-Pressed Silicon Nitride

The temperature dependence of the dielectric properties of hot-pressed silicon nitride varied considerably as a function of composition. The results at 35 GHz for samples with MgO at various concentrations added as sintering aids are shown in Figs. 15 and 16. In general, both the dielectric constant and loss tangent are smaller at lower MgO concentrations, with the 1% MgO sample (Ceradyne Corporation 147-A-3553-1) showing the best dielectric properties in terms of absolute magnitude as well as least variation as a function of temperature.

The results obtained at 35 GHz for samples with Y_2O_3 added as the sintering aid are shown in Figs. 17 and 18. As in the previous case, samples with lower Y_2O_3 concentrations exhibit lower values of the dielectric constant as well as loss tangent. In addition, measurements performed on the same sample with the electric field aligned parallel and perpendicular to the sample pressing axis indicate that the dielectric constant and loss tangent are less for the perpendicular orientation. There is also considerable variation in the loss tangent from sample to sample within a given sample type. As can be seen in Fig. 18, the measured loss tangent as a function of temperature for the 8% Y_2O_3 samples (Ceradyne Corporation, 147Y-1-3065) showed a difference by a factor of 2 between an average sample and one showing the least absorptive loss over the temperature range studied. The observed sharp increase in loss tangent at temperatures above 800°C is common to all samples, as is the general shape of the curve describing the increase in dielectric constant with temperature.

Results of measurements carried out at 94 GHz for the sample showing the least absorptive loss are shown in Figs. 13 and 14. As can be seen, the general behavior of the dielectric properties are similar to those observed at 35 GHz with the exception of the loss tangents being larger by a factor of 2 to 3 at temperatures below 800°C and increasing to a factor of 4 at higher temperatures.

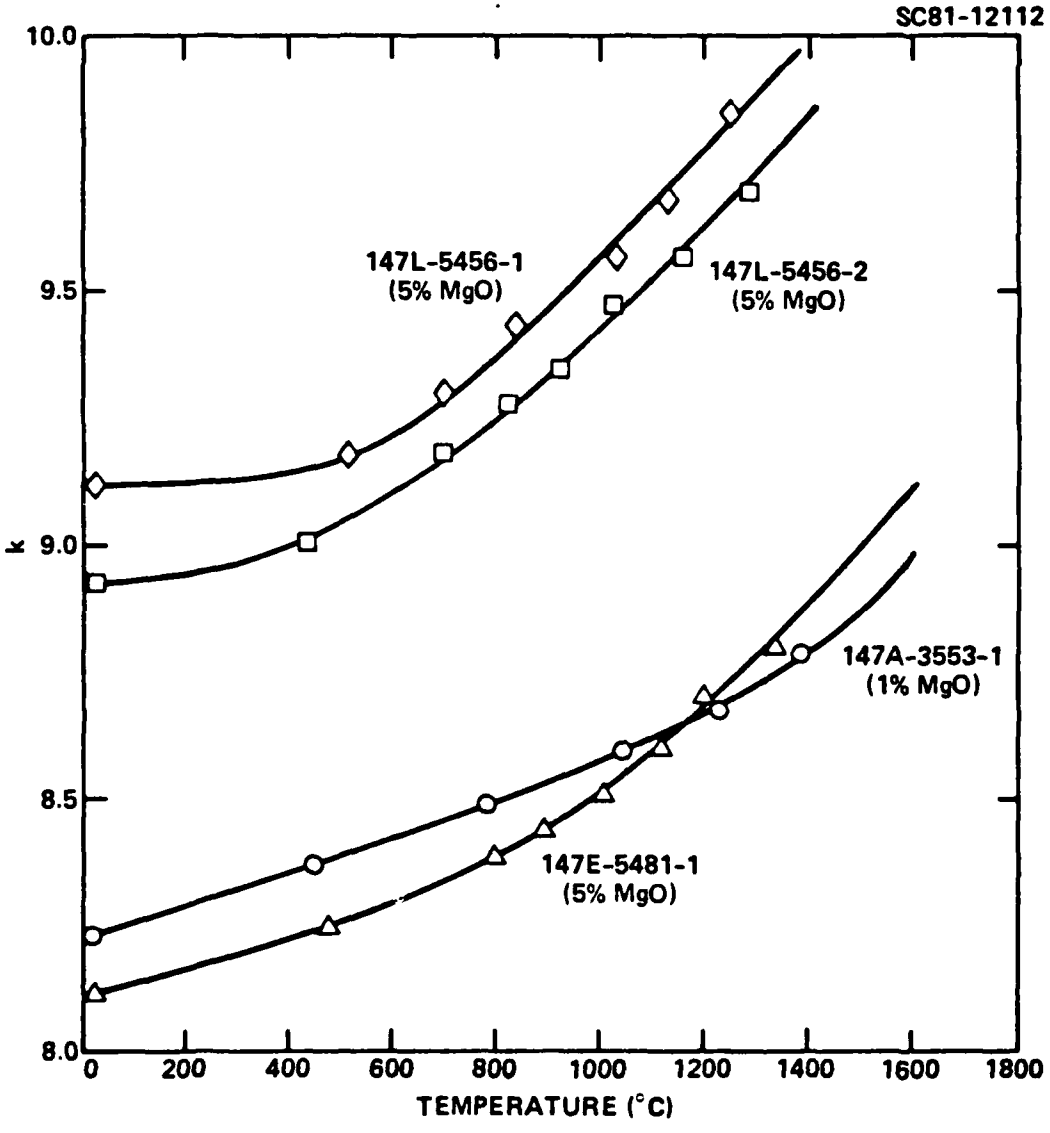

Fig. 15 Measured dielectric constant of hot-pressed silicon nitride (MgO as sintering aid) at 35 GHz.

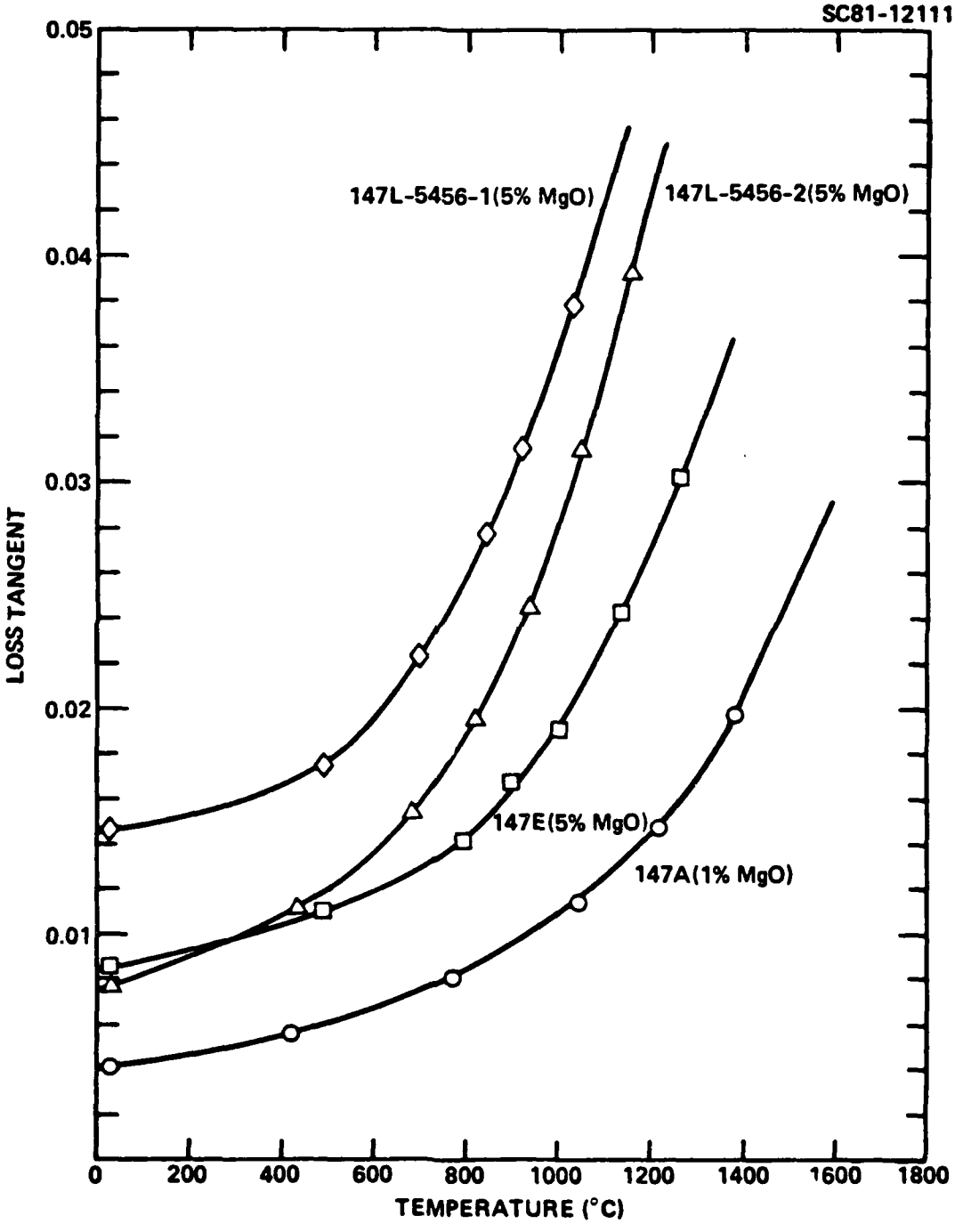

Fig. 16 Measured loss tangent of hot-pressed Si_3N_4 (MgO as sintering aid) at 35 GHz.

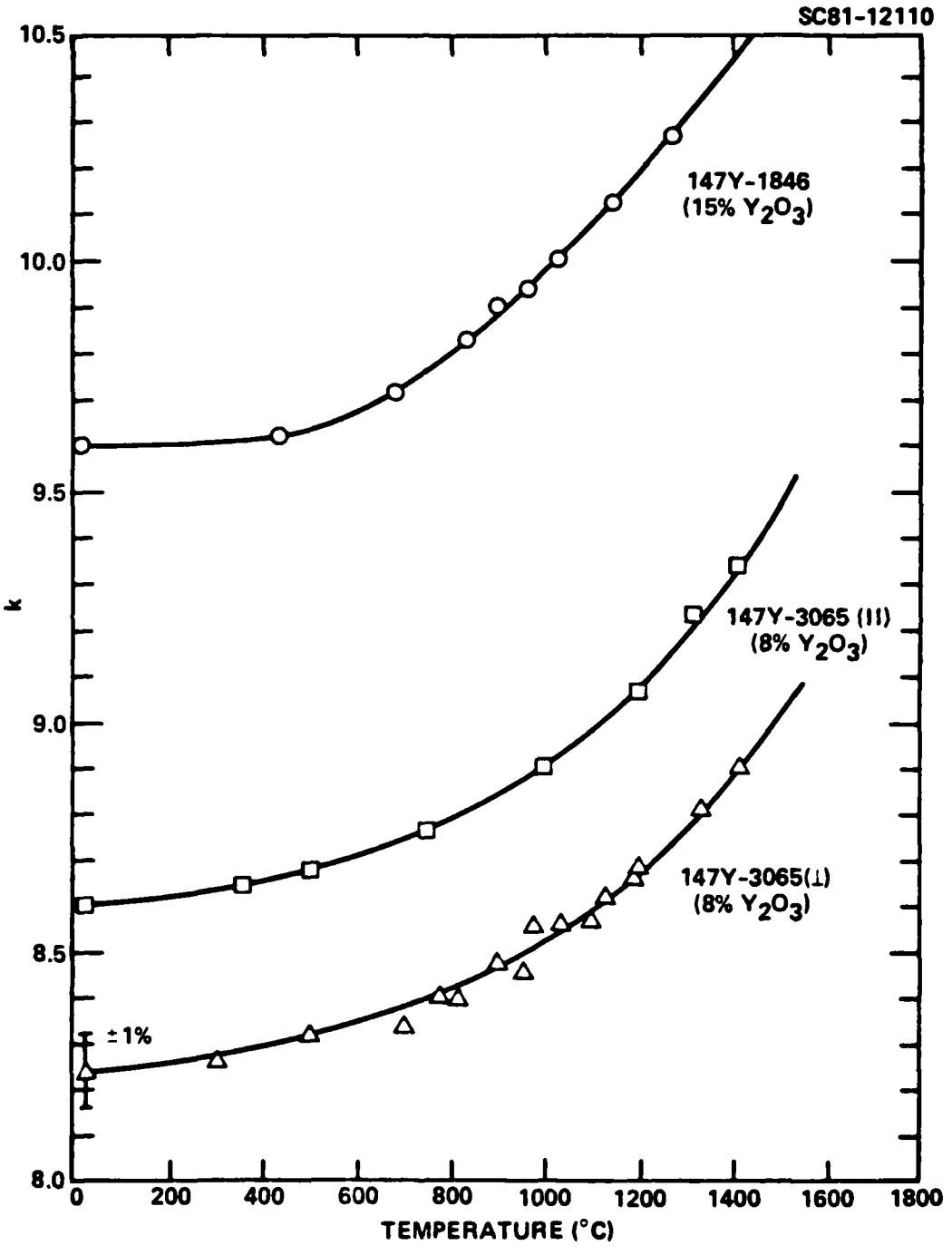

Fig. 17 Measured dielectric constant of hot-pressed Si_3N_4 (Y_2O_3 as sintering aid) at 35 GHz.

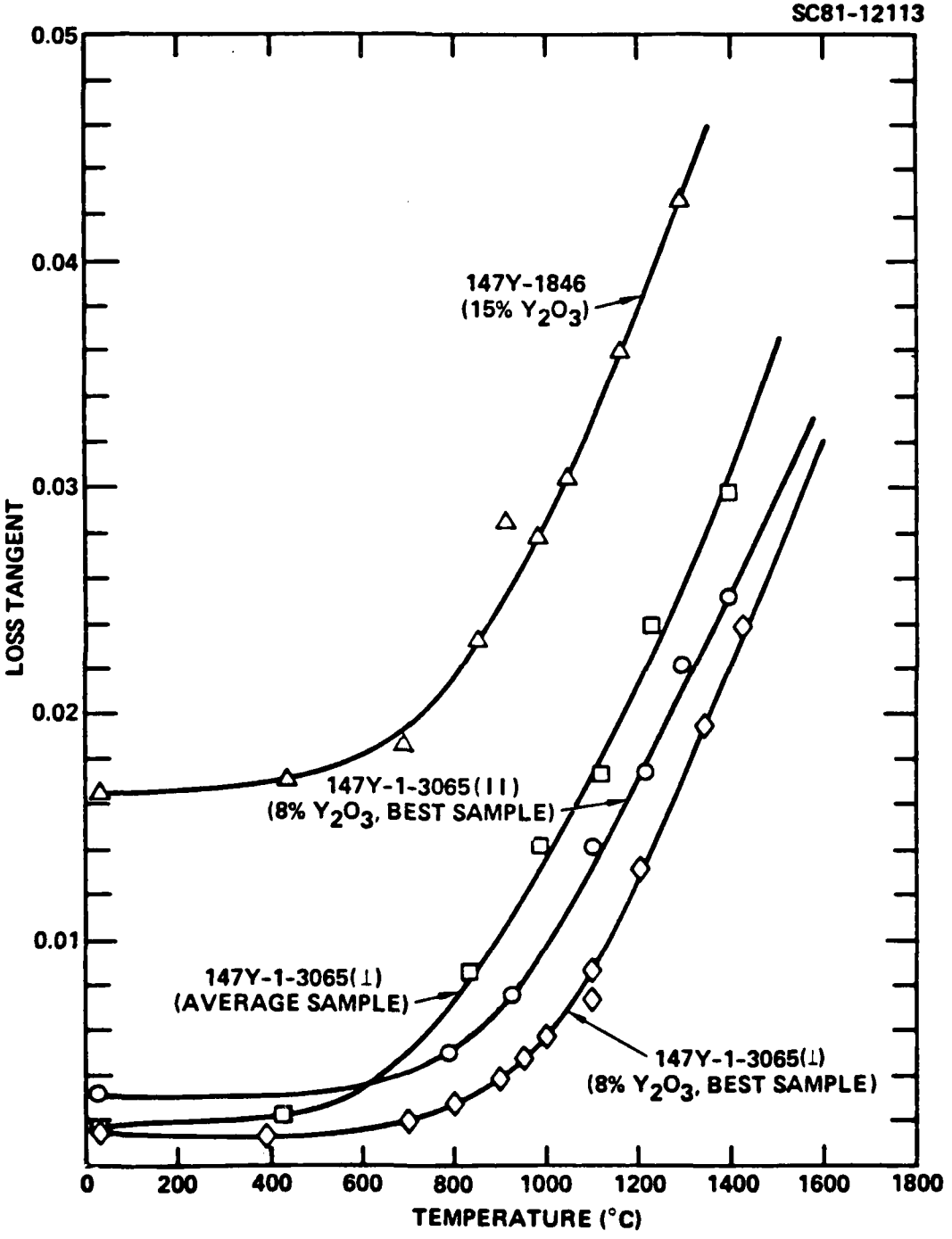

Fig. 18 Measured loss tangent of hot-pressed silicon nitride (Y_2O_3 as sintering aid) at 35 GHz.

8.0 CONCLUSIONS

The experimental measurement methods developed for determining the dielectric properties of solid samples at 35 and 94 GHz have been shown to be capable of generating the necessary temperature data for evaluating transmission properties of materials in millimeter wave radome applications. The measurement systems have been calibrated with fused silica and single crystal sapphire whose dielectric constant values span the range from 3.8 to 11.8. Experimental accuracy of better than 1% has been achieved over this range. Further verification of the absolute accuracy of the results has been accomplished with independent free-space transmission and reflection measurements. Loss tangent measurement accuracy better than ±10% with a detection limit of 0.0001 at 35 GHz and 0.0003 at 94 GHz has also been achieved.

Experiments carried out on BN, BeO, and various Si_3N_4 samples indicated that the measured dielectric constants for these materials are essentially independent of frequency between 35 and 94 GHz, whereas the loss tangents are approximately a factor of 2 to 4 larger at 94 GHz as compared to 35 GHz. For all samples studied, the dielectric constant was found to increase with temperature, with single crystal sapphire and beryllium oxide showing the largest increase and fused silica and boron nitride showing the smallest increase. The loss tangent of fused silica was found to be insensitive to temperature until its softening point is approached. For all other samples, there is a sharp increase in loss tangent values at temperatures higher than 800 - 1000°C.

The dielectric properties of hot-pressed silicon nitride were found to be strongly dependent on the composition and concentration of sintering aids. In general, better properties in terms of lower absorptive loss are observed for samples with lower concentrations of additives. Variations in dielectric properties for a given stated composition can be substantial and are most likely due to differences in the starting materials and the hot-pressing procedure used. Comparison of the results obtained for different samples indicated that the 8% Y_2O_3 samples (Ceradyne Corporation 147Y-1-3065) gave the best overall properties in terms of lowest loss and least variation of dielectric constant with temperature.

Thermal cycling tests indicated that non-reversible changes in dielectric properties with temperature could be present in ceramic materials but the magnitude of these changes is not large enough to be of significance in the determination of radome transmission properties for these materials. In addition, exposure of samples to water produced no changes in their dielectric properties once the surface water has been removed.

In terms of radome applications, the dielectric data obtained as a function of temperature indicated that all of the candidate materials studied have significant deficiencies at temperatures in excess of 1000°C. Although the boron nitride samples studied showed the best dielectric characteristics, their loss tangents still exceed 0.01 at temperatures above 1200°C. In addition, their mechanical and thermostructural properties are sufficiently poor that potential radome applications are limited to conditions where stresses and erosions are modest. Both the single crystal sapphire (and hence alumina) and BeO samples showed substantial variation of their dielectric constant with temperature, which make them unsuitable for radome applications over wide temperature conditions. Hot-pressed silicon nitride samples possess the best mechanical and thermostructural properties for high temperature radome applications. Their transmission characteristics, as deduced from the measured dielectric properties, are such that their performance at temperatures near 1000°C - 1200°C are unacceptable for most applications in excess of 1300°C. However, the results obtained for samples of varying formulation and composition strongly suggest that the observed dielectric properties are extrinsic to the material, i.e., primarily controlled by additives and impurities. It is, therefore, likely that substantial improvements can be made in their millimeter wave transmission properties if the materials parameters giving rise to the observed dielectric properties can be optimized to give the best overall performance in terms of mechanical and electromagnetic properties for specific radome applications.

9.0 RECOMMENDATIONS

The excellent mechanical and thermostructural properties, as well as ease of fabrication and relatively low cost, of hot-pressed silicon nitride (HPSN) have made it a primary candidate in advanced millimeter wave radar window applications for missile weapons systems where high temperatures caused by aerodynamic heating are expected. For example, the 147Y-1 type HPSN (8% Y_2O_3) samples (Ceradyne Corporation) studied in the current program have a mean flexure strength of 90,000 to 100,000 psi at room temperature and 70,000 psi at 1000°C, which is far superior to alternative radome materials. However, the millimeter wave dielectric properties obtained in this study indicated that the magnitudes of the increase in loss tangent and in the dielectric constant as a function of temperature for this material would cause significant transmission losses at high temperatures and, therefore, limit the usefulness of existing commercially available HPSN materials, in most radome applications, to below 1000°C. It is therefore highly desirable to identify the mechanisms controlling the observed dielectric properties in HPSN so that its transmission properties at high temperatures can be improved through optimization of materials and process parameters.

The dielectric properties of HPSN and their changes with temperature will affect the overall window transmission in several ways. An increase in the loss tangent will cause attenuation through the window, whereas changes in dielectric constant will result in mismatch of the window with the radar antenna system, resulting in increased reflective losses and beam distortion. As an example, if the thickness of a normal incidence HPSN radome is twice the half-wavelength of the microwave radiation in the material, (thickness ~ 0.3 cm at 35 GHz), the maximum allowable value for $\tan\delta$ corresponding to a 10% transmission loss is of the order of 0.01. Similarly, if the reflective losses are to be kept below 10%, the dielectric constant cannot vary by more than 8% over the desired operating temperature range. As the angle deviates from normal incidence, the relatively large value for k in HPSN causes the transmission to be strongly dependent on any changes in dielectric constant and, therefore, its variation with temperature needs to be further minimized.

The recent data obtained by Westphal[4], at 24 GHz on ultra-pure chemical vapor deposited (CVD) silicon nitride samples produced by G.E., indicate that the dielectric constant of these samples varies by less than 4% between 25 and 800°C, whereas the loss tangent was found to be below 0.0006 over the same temperature range. However, the thermostructural properties of CVD silicon nitride have not been fully established or optimized. In this respect, there appears to be an urgent need for extensive characterization of these properties for materials made by both CVD and HP methods so that their relative merits in terms of dielectric and strength properties at high temperatures can be compared.

The differences between the dielectric properties of CVD and hot-pressed silicon nitride have not been quantified in regards to specific mechanisms, but are most likely caused by composition variations, microstructural properties, and impurities. Recent studies carried out at the Science Center by F.F. Lange and D.R. Clarke[5,6] on various hot-pressed silicon nitride samples, using both MgO and Y_2O_3 as sintering aids, have indicated that, in addition to crystalline secondary phases, a residual glassy phase containing significant amounts of cation impurities are formed along the grain boundaries. The relatively low softening temperature of this impure glass contributes substantially to the degradation of mechanical properties of these samples at high temperature. It has also been observed that the cation concentration in the glassy phase can be reduced by exposure of the samples to oxygen at high temperatures. Under these conditions, the composition of the material is altered by an outward diffusion of cations and an inward diffusion of oxygen, and significant improvements in high temperature mechanical properties of the bulk material have been observed.

The presence of this glassy phase is likely to be the primary cause of the observed variation of millimeter wave dielectric properties at high temperatures, since it is expected that the dielectric constant and loss tangent of glass containing high cation impurities will rapidly increase as the softening temperature of the glass is approached. Consequently, if this can be demonstrated by a series of experiments correlating the observed dielectric properties of these materials with the volume fraction of the glassy phase and the

cation concentration, suitable fabrication processes can then be developed to minimize these effects for improving the millimeter wave transmission properties in these classes of materials. Aside from the previously mentioned oxidation procedure for lowering the cation concentration, it may also be feasible to reduce the presence of the glassy grain boundary phase itself by modifying the conditions under which the hot-pressing process is carried out. For instance, hot isostatic pressing techniques, whereby pressure in the region of 30,000 psi is used to lower the required pressing temperature by several hundred degrees centigrade, have been successfully utilized to prevent the formation of intergranular amorphous materials in iron-aluminum-silicon polyphase ceramics. Similar approaches could also prove to be effective in minimizing the glassy grain boundary phase in Si_3N_4 as well.

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Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172
HIGH TEMPERATURE MILLIMETER WAVE
CHARACTERIZATION OF THE DIELECTRIC
PROPERTIES OF ADVANCED WINDOW MATERIALS
William W. Ho

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Technical Report
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Key Words

Millimeter Wave Dielectric Properties
Antenna Window Materials

Final Report, September 1979 to January 1981

Experimental methods have been developed to determine the dielectric properties of candidate radome materials at 35 and 94 GHz for temperatures up to 1700°C. Measurements have been carried out on fused silica, single crystal sapphire, hot-pressed silicon nitride, beryllium oxide, and boron nitride as a function of temperature, composition and manufacturing procedures. Dielectric characterization data are presented in tabular and graphic forms to provide the necessary data base for evaluation of millimeter wave transmission properties of these materials in radome applications.

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